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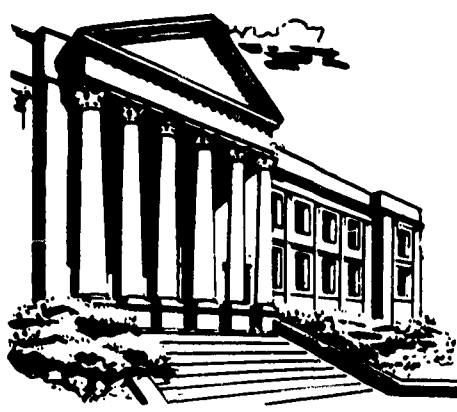


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Final Report

F-A2242

DEVELOPMENT OF ANTI-SEIZE
COMPOUND FOR TANK APPLICATION
PHASE II

by

Robert A. Erb

November 26, 1958 to November 25, 1960

Contract DA-36-034-ORD-2808-RD

Prepared for

RESEARCH AND DEVELOPMENT DIVISION
Ordnance Tank-Automotive Command
Detroit Arsenal
Center Line, Michigan

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ABSTRACT

The program for the continued development of the acrylic resin-tetrafluoroethylene-basic zinc chromate type anti-seize compound is described. Various experimental studies were undertaken including those of dispersion techniques; corrosion resisting properties, adhesion, suspension stability, brushmark flow-out, and other properties; application studies with dry-times and force-fit conditions. Samples of two different base compositions were submitted for field tests. As a result of the experimental program and field tests a modified FIL 92-46-1 composition (low acrylic resin content) is recommended for use.

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1. INTRODUCTION

This project for the further development of an anti-seize compound for use on tank vehicles is the continuation of the earlier program of 1957-1958 in which the basic Teflon-acrylic resin-corrosion inhibiting pigment system was developed. For information on the initial development of the system and on the physical properties under consideration, the Final Report for the previous project (Contract No. DA-36-034-ORD-2434) should be consulted.

The purpose of this present project has been to optimize the composition developed in the first phase through further experimental study of application and performance properties and to prepare specifications for its use. A variety of problems were encountered in this project including adhesion (particularly during assembly of parts and after long exposures to wet environmental conditions), brushmark leveling, corrosion resistance and resistance to conditions encountered in actual field testing. This report will cover our studies in approximately chronological arrangement.

2. EXPERIMENTAL PROGRAM

2.1 Dispersion Procedure Studies

The poor wettability of Teflon powder plus its poor behavior during milling procedures led to an experimental study of a variety of mixing sequences and dispersion techniques (three-roll milling, ball milling, etc). The study was extended to include variations in the grinding vehicles and diluents and their effect on the suspension stability of the milled concentrate and on the fineness and uniformity of the dispersion. Measurements were made of the contact angles of 12 solvents and resin solutions on Teflon plates; these ranged from 9° for heptane to 53° for a 50% solution of Acryloid B-72 in xylene. The solvent contact angle was considered in conjunction with its surface

tension, solubility parameter and solvency for Acryloid B-72. MIBK (methyl isobutyl ketone) had the best wetting properties (14° contact angle) for the Teflon among any of the good Acryloid solvents studied. The best dispersion procedure found in a series of tests was, using MIBK as the sole dispersion solvent, to wet the Teflon powder with the solvent only, dissolve the Acryloid in the Teflon-solvent slurry, stir in the zinc chromate, and mill the entire mixture for one or two passes through a very tight three-roll mill. The dispersion thus obtained was superior to the others studied at the same time in hot-cold cycling stability and in draw down smoothness.

Filtering the mill paste through an open cloth mesh was found to be useful in preventing the occurrence of skins and flakes of dried material in the final composition.

2.2 Systematic Solids Proportions Studies

An experimental program was undertaken in February 1959 to systematically vary the proportions in the ternary system — basic zinc chromate - Teflon 7 - Acryloid B-72 — and study the effects of such variations on product properties. A triangular graph was prepared with 12 compositions plotted thereon, ranging from 22 to 52% basic zinc chromate and 11 to 21% Teflon by weight in the solids. These were all made with properties such as Brookfield viscosity, hot-cold cycling stability and separation on standing recorded. It was noted that suspension stability increased with an increase in the proportion of particulate material (Teflon and zinc chromate) in the solids content.

2.3 Incorporation of Suspension Stabilizers

The use of suspension stabilizers was investigated at this point. Three commercial gelling agents were studied: Bentone 34 (a montmorillonite derivative by National Lead Company), Cab-O-Sil M-5 (ultrafine silica by Godfrey L. Cabot, Inc.) and Attagel (an attapulgite derivative). All three produced substantial improvements in suspension

stability. Also tried later was Bentone 38, another montmorillonite derivative. The most satisfactory agent from over-all considerations was Bentone 34. The triangular graph composition FIL 92-37-5 modified with two parts of Bentone 34 was designated as FIL 92-42-1, and later, in a large batch, as FIL 92-46-1. (This was subsequently studied in field tests at Yuma.)

2.4 Corrosion Tests on Triangular Graph Formulations

Salt-spray corrosion tests were run on seven of the triangular graph systematic compositions plus the three with gelling agents as modifications of FIL 92-37-5. After one 200 hour test with sprayed coatings, in which corrosion trends were difficult to identify, a second test was set up with both brushed and sprayed panels, with randomized position-changing throughout the test. The over-all observations for this test are shown in Table 1. In general, for a given composition, the brushed panel resisted corrosion better than the sprayed panel. The corrosion was followed on two sides (arbitrarily designated + and -) of the sprayed panels. The rankings given in the last column for the brushed panels were based on consideration of the properties listed, along with over-all appearance.

At this point we analyzed the relationship of corrosion to composition. On Graph 1 were plotted the compositions tested. Line A was drawn and it was pointed out that the six compositions ranked best were on the high-Acryloid side of Line A, whereas, the four worst were all on the low-Acryloid side of Line A. The conclusion drawn, which appears to be valid for exposed panel corrosion tests, was that the higher-Acryloid compositions were most suitable in this respect. The X's at the top of Graph 1 were drawn to indicate regions of interest for the subsequent formulation program.

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Table 1

RESULTS OF THE 192 HOUR SALT SPRAY TEST

| Formula Number | Composition of Mill Paste, % By Weight | | | | | Method of Application | Dry Film Thickness (mils) | | Texture of Coating | Blistering | | Peeling (Both Sides) |
|----------------|--|----------|---------------------|-----------------------|-------|-----------------------|---------------------------|------------|--------------------|--------------------------|-----------------|----------------------|
| | Acryloid B-72 | Teflon 7 | Basic Zinc Chromate | Suspension Stabilizer | MIBK | | + Side | - Side | | + Side | - Side | |
| 92-37-1 | 21.63 | 12.29 | 24.56 | - | 41.52 | Brush Spray | 2-2.5 1.5 | - 1-1.5 | Smooth Smooth | Mod. Heavy Mod. Heavy | - Mod. Heavy | None None |
| 92-37-3 | 26.60 | 6.20 | 23.77 | - | 43.41 | Brush Spray | 2-2.5 2-2.5 | - 2 | Smooth Smooth | Heavy Mod. Heavy | - Moderate | None None |
| 92-37-4 | 26.23 | 11.73 | 17.85 | - | 44.19 | Brush Spray | 2-2.5 2.5-3 | - 3-3.5 | Smooth Rough | Mod. Heavy Heavy | - Heavy | None None |
| 92-37-5 | 28.55 | 8.78 | 17.57 | - | 45.10 | Brush Spray | 2-2.5 2-2.5 | - 2 | Smooth Smooth | Moderate Heavy | - Heavy | None None |
| 92-37-7 | 30.40 | 11.20 | 11.74 | - | 46.66 | Brush Spray | 2 2-2.5 | - 2 | Smooth Smooth | Slight Mod. Heavy | - Moderate | None None |
| 92-37-9 | 34.68 | 5.69 | 11.39 | - | 48.24 | Brush Spray | 2-2.5 3.5-4 | - 3-3.5 | Smooth Rough | V. Slight Mod. Heavy | - Mod. Heavy | None None |
| 92-37-11 | 19.32 | 9.66 | 31.39 | - | 39.63 | Brush Spray | 2-2.5 4 | - 3 | Smooth Rough | Heavy Heavy | - Heavy | None None |
| 92-42-1 | 28.55 | 8.78 | 15.57 | 2.00* | 45.10 | Brush Spray | 2-2.5 2-2.5 | - 1.5-2 | Smooth Smooth | Slight Heavy | - Heavy | None None |
| 92-42-2 | 28.55 | 8.78 | 15.57 | 2.00** | 45.10 | Brush Spray | 2-2.5 - | - - | Smooth - | Slight - | - - | None None |
| 92-42-3 | 28.55 | 8.78 | 15.57 | 2.00*** | 45.10 | Brush Spray | 2-2.5 2-5 | - 1.5-2 | Smooth Smooth | Slight Heavy | - Mod. Heavy | None None |

* Bentone 34
** Cab-O-Sil
*** Attagel 20

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Table 1

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| Formula Number | Composition of Mill Paste, % By Weight | | | | | Method of Application | Dry Film Thickness (mils) | | Texture of Coating | Blistering | | Peeling (Both Sides) |
|----------------|--|----------|---------------------|-----------------------|-------|-----------------------|---------------------------|------------|--------------------|--------------------------|-----------------|----------------------|
| | Acryloid B-72 | Teflon 7 | Basic Zinc Chromate | Suspension Stabilizer | MIK | | + Side | - Side | | + Side | - Side | |
| 92-37-1 | 21.63 | 12.29 | 24.56 | - | 41.52 | Brush Spray | 2-2.5 1.5 | - 1-1.5 | Smooth Smooth | Mod. Heavy Mod. Heavy | - Mod. Heavy | None None |
| 92-37-3 | 26.60 | 6.20 | 23.77 | - | 43.41 | Brush Spray | 2-2.5 2-2.5 | - 2 | Smooth Smooth | Heavy Mod. Heavy | - Moderate | None None |
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| 92-42-2 | 28.55 | 8.78 | 15.57 | 2.00** | 45.10 | Brush Spray | 2-2.5 - | - - | Smooth - | Slight - | - - | None None |
| 92-42-3 | 28.55 | 8.78 | 15.57 | 2.00*** | 45.10 | Brush Spray | 2-2.5 2-5 | - 1.5-2 | Smooth Smooth | Slight Heavy | - Mod. Heavy | None None |

* Bentone 34
** Cab-O-Sil
*** Attagel 20

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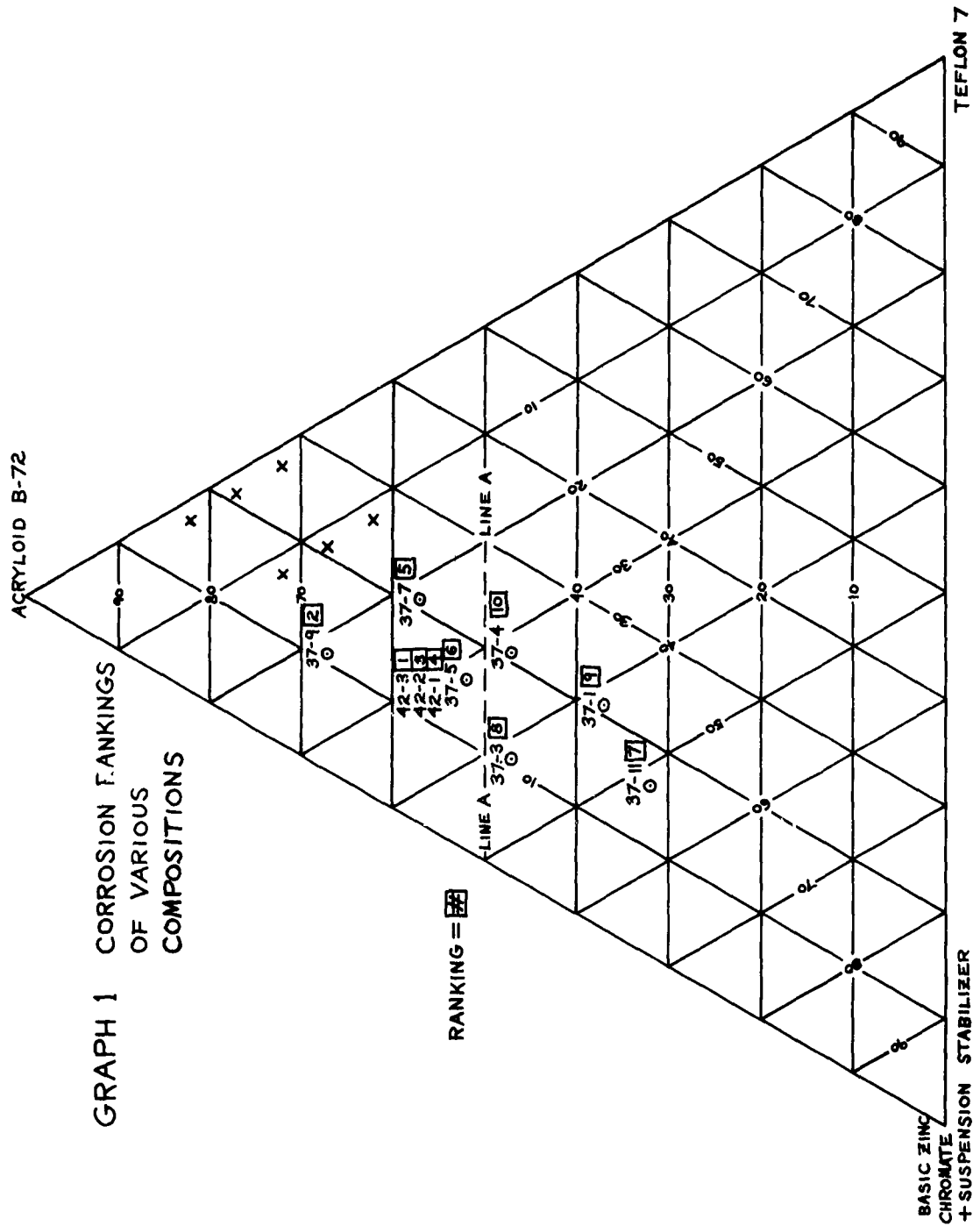
Table 1

RESULTS OF THE 192 HOUR SALT SPRAY TEST

| Results of the Corrosion Test Exposure | | | | | | | | | | |
|--|--------------------------|------------|------------|-------------------------|--|--------------|--|------------|------------------------------------|--|
| Film thickness (in.) | Texture of Coating | Blistering | | Peeling (Both Sides) | Discoloration (Whitening Due to Loss of Zinc Chromate) | | Pitting of Metal (Generally Under Blisters) | | Rusting under Intact Film | Ranking of Corrosion Protection Based on Brushed Panels |
| | | + Side | - Side | | + Side | - Side | + Side | - Side | | |
| - | Smooth | Mod. Heavy | - | None | Little | - | Moderate | - | Nil | 9 |
| 1-1.5 | Smooth | Mod. Heavy | Mod. Heavy | None | V. Little | Little | Mod. Heavy | Mod. Heavy | Nil | |
| - | Smooth | Heavy | - | None | Little | - | Mod. Heavy | - | Nil | 8 |
| 2 | Smooth | Mod. Heavy | Moderate | None | Some | Little | Moderate | Moderate | Nil | |
| - | Smooth | Mod. Heavy | - | None | Some | - | Mod. Heavy | - | Nil | 10 |
| 3-3.5 | Rough | Heavy | Heavy | None | Little | Some | Moderate | Mod. Heavy | Nil | |
| - | Smooth | Moderate | - | None | V. Little | - | Moderate | - | Nil | 6 |
| 2 | Smooth | Heavy | Heavy | None | Some | Some | Mod. Heavy | Mod. Heavy | Nil | |
| - | Smooth | Slight | - | None | Little | - | V. Slight | - | Nil | 5 |
| 2 | Smooth | Mod. Heavy | Moderate | None | Some | Little | Slight | V. Slight | Nil | |
| - | Smooth | V. Slight | - | None | V. Little | - | Nil | - | Nil | 2 |
| 3-3.5 | Rough | Mod. Heavy | Mod. Heavy | None | Little | V. Little | Moderate | Moderate | Nil | |
| - | Smooth | Heavy | - | None | Some | - | Heavy | - | Nil | 7 |
| 3 | Rough | Heavy | Heavy | None | Some | Considerable | Heavy | Heavy | Nil | |
| - | Smooth | Slight | - | None | Little | - | Slight | - | Nil | 4 |
| 1.5-2 | Smooth | Heavy | Heavy | None | Considerable | Considerable | Heavy | Heavy | Nil | |
| - | Smooth | Slight | - | None | Little | - | Nil | - | Nil | 3 |
| - | - | - | - | None | - | - | - | - | Nil | |
| - | Smooth | Slight | - | None | V. Little | - | Nil | - | Nil | 1 |
| 1.5-2 | Smooth | Heavy | Mod. Heavy | None | Considerable | Considerable | Heavy | Mod. Heavy | Nil | |

* Bentone 34
** Cab-O-Sil
*** Attagel 20

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2.5 Experimental Compound to Yuma

A gallon of experimental anti-seize compound, FIL 92-46-1, was made, and shipped to the U. S. Ordnance Test Activity Yuma, Arizona on 14 April 1959 for field testing. The formulation and cost of the mill paste is shown below. In use, this paste was diluted about 1:1 by volume with methyl isobutyl ketone, which brings the raw material cost of the finished product down to \$4.28 per gallon.

| <u>Ingredient</u> | <u>% by Weight</u> | <u>Cost per Pound*</u> | <u>Cost per 100 Pounds of Compound</u> |
|------------------------|--------------------|------------------------|--|
| Acryloid B-72 | 28.55 | \$0.81 | \$23.12 |
| Basic zinc chromate | 15.57 | 0.34 | 5.29 |
| Teflon 7 | 8.78 | 4.60 | 40.39 |
| Bentone 34 | 2.00 | 0.60 | 1.20 |
| Methyl isobutyl ketone | <u>45.10</u> | 0.145 | <u>6.54</u> |
| | 100.00 | | 76.55 |

Raw-material cost of base stock per pound: \$0.7655

Specific gravity of base stock: Approx 1.15 (9.6 lb/gallon)

Raw-material cost of base stock per gallon: \$7.35

* April 1959 prices

Some properties of the shipped batch of FIL 92-46-1 (mill paste) were as follows:

| | | |
|------------------------------|------------------|-----|
| Initial Brookfield viscosity | 6 RPM | 190 |
| at 25°C (poises): | 12 RPM | 108 |
| | 30 RPM | 80 |
| | 60 RPM | 60 |
| Specific gravity: | 1.175 | |
| Measured solids (by wt): | 58.5% | |
| One month separation | 0% | |
| One month pourability | Freely pourable | |
| Four month separation | 1% clear liquid | |
| Four month pourability | Remains pourable | |

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In the making of this sample, two final three-roll mill passes were made with the entire batch, which was then pressure-filtered through a 100 mesh wire screen. Instructions for surface preparation and application were sent to Yuma along with safety precautions.

A different batch of experimental anti-seize compound with the same composition and designated FIL 92-46-1A was mailed to Detroit Arsenal on 20 May 1959.

2.6 Additional Triangular Graph Corrosion Tests

The six compositions indicated by the X's on Graph 1 were made and tested on panels in the standard salt-spray test. The corrosion resistance provided by these compositions on the panels was even better than the previously-mentioned series. (A control coating of unmodified Acryloid B-72 included, however, was seen to be poor with heavy blistering and pits under the blisters.) Examination was made at 192 and 500 hours. The only adverse phenomena of note were the formation of water-filled blisters on all samples and the presence of water films between the coating and the substrate (i.e., complete loss of adhesion over much of the film area). These phenomena were overshadowed at the time by the excellent corrosion results. They became objects of substantial further experimental study, however, and eventually became part of the basis for rejecting the high-Acryloid system in favor of low-Acryloid content, as will be seen later.

2.7 Experimental Compound to Aberdeen

Two quarts of experimental anti-seize compound FIL 15-13-2 was made, and shipped to Aberdeen Proving Ground, Aberdeen, Maryland on 31 July, 1959. This is a high-Acryloid composition of the following percentage composition by weight (base stock): Acryloid B-72 (100% solids), 36.28; Teflon 7, 8.05; Basic zinc chromate, 6.05; Bentone 34, 2.00; MIBK, 47.64. This composition had the solids proportions equivalent to the best compound in the 500 hour corrosion test series.

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2.8 Soil Burial Test Start

An alkaline soil burial test was started on August 18, 1959 with standard-torqued nut-bolt combinations and panels, on which coatings of anti-seize compounds had been applied. This test was continued for 14 months and withdrawn in October 1960; the results are described in a later section.

2.9 Yuma Field Test Results

The Franklin Institute received its first report September, 1959 of the Yuma tests for the 1000 and 2000 mile inspections, listed as: w. Anti-seize Compound (IT-5161/Y339); from, Third Memorandum report on test of Components for M48A2 Tank, Projects IT-5137 through IT-5150/Y339 (10 July through 5 August 1959). A final report covering the 1000, 2000, and 4000 mile inspections was received in June 1960. This extract from Report No. OTA/IT-5057/11 is reported in its entirety as follows:

3.17 Anti-Seize Compound (4000 miles, 9B0665)

Anti-Seize Compound was applied to the suspension components on the right side of one vehicle (torsion bar serrations, anchor blocks, track pins, end connectors, sprocket-to-hub-to-drive shaft) to aid in disassembly of mating steel parts. The metal surfaces were first wire brushed, thoroughly removing all the rust, then washed with a clean cloth soaked with stabilized trichloro-ethylene (Military Specification MIL-T-7003) to remove all grease.

3.17.1 One-Thousand Mile Inspection

The 1000 mile inspection of the end connectors and track pins revealed that approximately twice as much energy was required to break the initial set of the untreated end connectors. Once the initial set had been broken, the efforts required to completely remove the connectors from the treated and untreated link pins was about the same. The treated

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surfaces were found to be mildly corroded with slight peeling of the anti-seize coating whereas the untreated surfaces showed extensive corrosion.

3.17.2 Two-Thousand Mile Inspection

The 2000 mile inspection of the torsion bars (both splined ends) revealed the untreated torsion bars were easier to remove than the treated bars. A close inspection indicated the anti-seize coating decreased the tolerance between the end of the torsion bars and the torsion bar anchors causing a forced fit. This damaged the coating surface allowing the bars and anchors to become slightly corroded.

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Inspection of the end connectors and track pins at this time indicated a slight peeling of the anti-seize coating and slight corrosion of the surfaces. Replacement of the end connectors and track was necessary during this inspection.

3.17.3 Four-Thousand Mile Inspection

Final inspection of the treated surfaces of the torsion bars, end connectors, track pins, sprocket-to-hub and hub-to-drive shaft surfaces indicated the treated surfaces were slightly corroded with slight peeling of the coating, while the untreated surfaces showed extensive corrosion.

CONCLUSIONS

_____ Anti-Seize Compound was considered satisfactory.

We were interested to see that "approximately twice as much energy was required to break the initial set of the untreated end

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connectors" as for the end connectors treated with the FIL anti-seize compound. We are particularly gratified with the over-all conclusion, "anti-seize compound was considered satisfactory."

We were concerned, however, that the anti-seize compound was removed to an extensive degree in the force fit situation (2000 mile inspection) for the torsion bars and anchors. The experimental program described in the next section was directed toward this problem.

Another point of note is that, at the 2000 mile inspection, the untreated torsion bars were easier to remove than the treated bars. This behavior was repeated to a severe extent by the unsatisfactory FIL 15-13-2 anti-seize compound tested later at Aberdeen. It could be suggested that in cases where tight metal-to-metal contact is not present during a simple insertion-removal situation and where very large areas are involved in simple shear-type motion (both of which conditions occur in the splined end fitting) of the torsion bar and anchor block, a solid film anti-seize compound of substantial thickness (e.g., 1 mil) would as a rule increase the removal force needed compared to that with an untreated bar. (This, of course, might not hold true where gross corrosion, fretting or other types, would occur.) It is suggested that if low removal forces are required for torsion bar removal, an easily-deformable type compound should be considered such as a molybdenum disulfide-containing grease. Such a system, of course, does not fulfill a number of the product requirements under the present contract (e.g., sand abrasion resistance).

2.10 Force-Fit Tests

Because the Acryloid-Teflon-zinc chromate anti-seize compound in the previous contract program had shown pronounced resistance to removal under force-fit situations after thorough drying (e.g., 24 hours), the FIL 92-46-1 compound previously sent to Yuma was studied in an experimental program. In this study a 1.000 inch diameter drill-stock rod and a 1.002 inch inside-diameter bushing were used as a force-fit device

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having a 1 mil annulus. One coat of FIL 92-46-1 diluted 1:1 by volume with MIBK was applied to the rod only, and allowed to dry. The dry film thickness was measured by vernier calipers and a Tinsley magnetic gauge and found to be approximately 2 mils. The sample was stored for one week in air before the rod was forced into the bushing. The coating deformed during the assembly with a very small amount of build-up at the forward edge of the bushing. Examination after disassembly showed that (as hoped) no removal to the bare metal occurred; in some areas the anti-seize compound thickness on the rod surface was sheared to 0.5 mil. (Good adhesion to the substrate for this system was confirmed by the Scotch Tape Test.) In some areas of the inner surface of the bushing, a film of the compound was attached. The forces involved in assembly and disassembly were as follows: on, 1280 to 2400 lb; off, 900 to 2000 lb. These results were in line with those obtained much earlier with the basic Teflon-Acryloid-zinc chromate compound.

As the Yuma test results with the torsion bars did not duplicate the above performance, consideration was given to the possibility that the drying time difference prior to assembly (30 minutes in the instructions to Yuma, 7 days in the above test) was the cause of the substantial difference between expected and observed behavior. The force-fit test was rerun with the same compound in a 2 mil thickness on the same rod-bushing assembly, but with only a 30 minute driving period before testing. This time, substantial amounts of the coating were scraped off and piled up the the front edge of the bushing; examination of the rod surfaces after disassembly showed large areas bared to the metal. Protection in such a system would be greatly diminished, particularly with regard to ordinary and fretting corrosion.

On-off force measurements were made with two 30 minute dry time samples:

On, 600 lb; off, immediately, 200 lb.

On, 600 lb; off, after two days, 400 lb.

These are lower values than for the well-dried sample. For assembly, this low value may be due to a reduced cohesive and adhesive strength of the sample. For disassembly, the low values may stem from the same reasons as well as from the discontinuities caused by removal of the compound from some areas in the annulus. It is noted that the resistance to removal rose after two days standing in a non-corrosive environment.

A photograph of a 30 minute dry time force-fit test is shown in Figure 1. The same rod and bushing, but with a 3 day dry time sample, is shown in Figure 2. (The 3 day sample behaved similarly to the 7 day sample mentioned previously.) The compound is FIL 92-46-1 in both figures.

The pronounced superiority of the samples which dried for several days over the 30 minute dry time samples prompted consideration of two approaches.

The first approach involved the study of the use of more volatile solvents in place of all or part of the MIBK. In this study, solvents were chosen which had high evaporation rates and good solvency for Acryloid B-72. Comparative dispersions were made using as dispersion solvents: (1) MIBK, (2) acetone, (3) ethyl acetate, and (4) ethylene dichloride. The same solvents were also used in the letdowns, including each of the other three with the MIBK dispersion. Steel panels were coated with each composition and adhesion tests (Scotch tape test) were run after 30 minutes and also longer periods (e.g., 24 hours). To summarize the results: with dry times of more than 30 minutes MIBK was the best solvent with respect to adhesion produced. With short dry times (30 minutes or less), no significant differences in adhesion were found among the solvents. Thus, simply switching to more volatile solvents did not serve to permit shortening the dry time before assembly. This is in spite of experimentally measured decreases in set-to-touch and dry-through times through the use of the more volatile solvents, and probably is due to the reduced ultimate adhesion level attainable with the more volatile solvents.

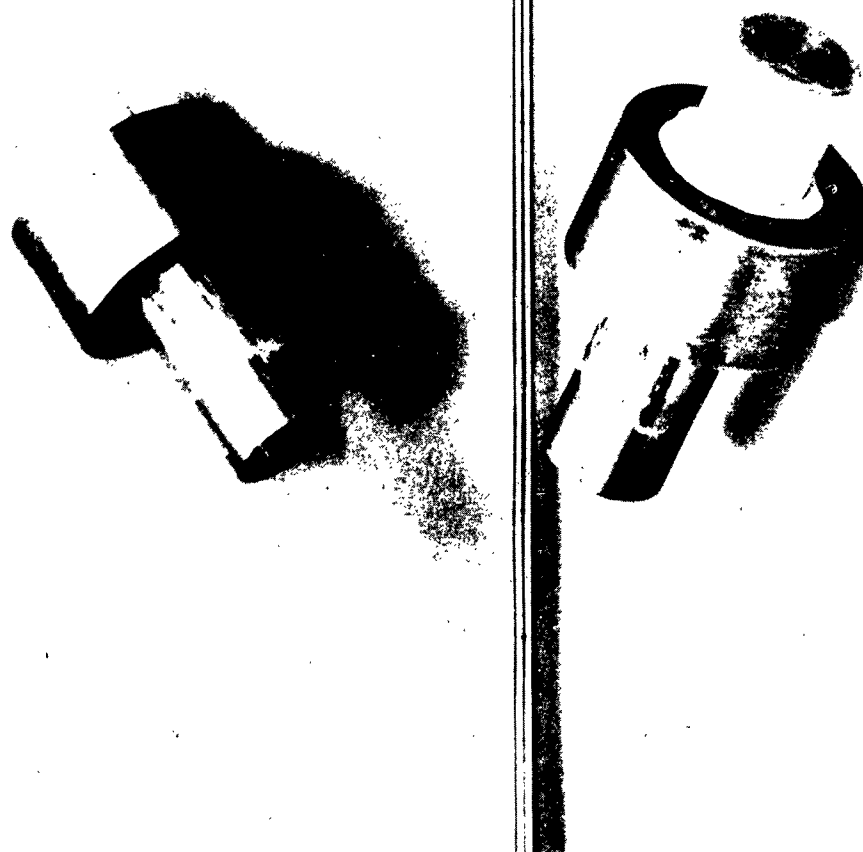


FIGURE 1. FORCE - FIT TEST OF ANTI-SEIZE COMPOUND (FIL 92-46-1) AFTER

30-MINUTE DRY TIME

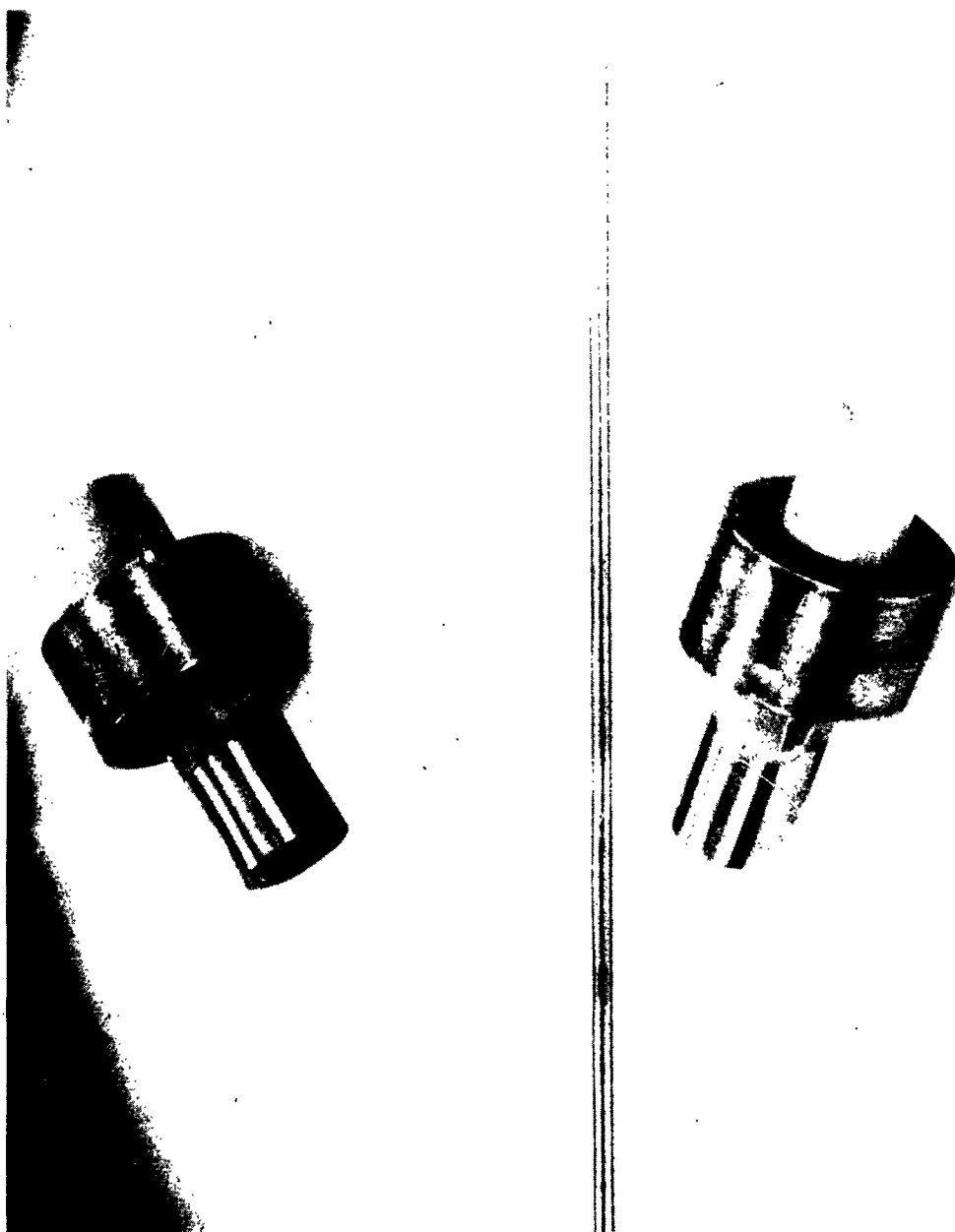


FIGURE 2. FORCE-FIT TEST OF ANTI-SEIZE COMPOUND (FIL 92-46-1) AFTER
3-DAY DRY TIME

The second approach is simply to increase the drying time specified for the compound in use. A step in this direction was taken when the application instructions for the FIL 15-13-2A experimental compound submitted to Aberdeen on November 16, 1959 for field test results (not back yet) were set up so that the coating for torsion bar splines was to dry for 2 hours before assembly, with the 30 minute dry-time stipulation for the other parts retained. That this 2 hour dry-time is probably insufficient for the difficult force fit applications is suggested from: (1) the force fit tests as pictured above; (2) the field test results with FIL 92-46-1 and 15-13-2 compounds. From these considerations, the drying time to be specified before assembly of parts is being set at two days (48 hours). Shorter times, of course, could be used in emergencies; however, poorer performance would have to be expected. These relatively long air-dry periods strongly suggest factory application to parts before use. (Baking to shorten the drying time could probably not be used in the field but might be considered where such facilities are available.) Another consideration here is that a slowly evaporating solvent has more recently been made part of the diluent (see Sec. 2.13).

2.11 Preliminary Specifications

In the December, 1959, monthly progress report a preliminary quality control specification for the anti-seize compound (polytetrafluoroethylene and zinc chromate filled resin type) was set forth. This has served as a basis for the specification included as Appendix C to this report.

2.12 Resin Modifications

In an effort to improve the over-all adhesion characteristics of the system (with particular emphasis on the FIL 15-13-2 composition being studied at the time) an experimental investigation was made of the use of film-forming resins to modify the Acryloid B-72. The first experiments in unpigmented systems with two resins chosen for their

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literature-described adhesion properties indicated some improvement of dry-film adhesion. The modifying resins were Flexalyn (Hercules diethylene glycol ester of rosin) and Vinylite AYAA (Union Carbide vinyl acetate polymer). Pigmented formulations were also made with each resin, with a 1:9 substitution for Acryloid B-72 in the FIL 15-13-2 composition. The Flexalyn in this case appeared to increase the adhesion slightly and the AYAA decrease it slightly, as measured by the Scotch tape test.

A larger scale test was conducted with three modifying resins: Flexalyn, Flexalyn C (another rosin derivative) and Paraplex G-50 (a polyester plasticizer by Rohm & Haas). Each was included in a base composition in two concentrations, equal to 3.6% and 7.2% of the total solids content. In this program, controls (92-46-1 and 15-13-1 compositions) were also included. An extensive testing program was run on these compounds including the following tests: Brookfield and Zahn Cup viscosity; fineness of grind; sand abrasion coefficient; metal adhesion by knife test; metal adhesion by tape test; and salt-spray corrosion test with 200 and 400 hour examination (notations for blistering, discoloration, relative adhesion, general rust, pitting, and corrosion extent for both brushed and sprayed panels). Some observations and conclusions are as follows:

(1) In the dry-film adhesion tests the modifying resins in no case improved the adhesion and in the higher proportions decreased the adhesion in each case.

(2) In the salt-spray test the adhesion was virtually lost completely with the high-Acryloid compounds (i.e., in every sample except the FIL 92-46-1 type), with water-filled blisters also being very prevalent with these materials.

(3) The FIL 92-46-1 type compound ranked highest in over-all coating appearance as well as in dried-out coating adhesion after the 400 hour salt-spray test.

(4) The FIL 92-46-1 film had a considerably lower sand abrasion resistance than did the higher-Acryloid coatings (3.8 liters/mil vs. 6.3 to 16.6 liters per mil); this level of 3.8 liters/mil should be satisfactory for the compound use purposes, however.

The most important result of these resin-modification studies appears to be the definite advantage in adhesion after exposure to a "wet" environment of the low-Acryloid 92-46-1 system over the high-Acryloid systems (resin modifications being inconsequential).

2.13 Brushmark Flow-out

Brushing as a method of applying the coatings has shown superiority over spraying in the corrosion results of several salt-spray test series. However, these Teflon-containing dispersions have as a rule shown a pronounced tendency to retain brushmarks, even when well-diluted to promote flowing. It is suggested that the problem is aggravated by the poor wettability of the Teflon and its relatively large particle size.

A program in April, 1960, was aimed at improving the brushing qualities of the anti-seize compositions. The approach taken was to incorporate in the composition as diluted for brushing a slow-evaporating liquid which is a very good solvent for the resin content. In such a case the diluents, cosolvents or poorer solvents in general evaporate first and the strong solvent remaining permits viscous flow without a gelling action. The evidence is that if slower evaporating diluents (e.g., hydrocarbons) are used, gelling occurs early during the drying process, thereby prohibiting brushmark flow-out.

The most satisfactory solvent found in our studies for the promotion of brushmark flow-out is ethylene glycol monoethyl ether (Cellosolve, Union Carbide Chemicals Company). This solvent is included in the diluting solvent composition for the recommended anti-seize compound formulation.

2.14 Study of Pigments Other Than Basic Zinc Chromate

In the salt-spray corrosion test studies with the high-Acryloid compositions, water-blister formation and adhesion loss were common phenomena; this was postulated as due to combination of a highly-impervious film with the basic zinc chromate pigment, which has relatively high water solubility (among pigments) and is reported in the literature as sometimes inducing water-blister formation. In the lower-resin material, such as 92-46-1, the higher pigment content with the higher porosity partly induced by leaching of the pigment apparently tends to prevent differential pressure build-up with resulting adhesional failure.

A program was initiated to find a suitable low-solubility corrosion-inhibiting pigment that would not induce water-blister formation or adhesional failure, to replace the basic zinc chromate. The literature was studied and manufacturers of corrosion-inhibiting pigments were contacted to find a number of such pigments for which an experimental program would be of value. After this study the following were chosen: 1) basic zinc chromate (as a control); 2) chrome yellow medium (lead chromate); 3) zinc yellow; 4) basic lead silico chromate; 5) basic lead chromate; 6) strontium chromate.

For each of the above pigments three solids compositions were chosen on an Acryloid-Teflon-pigment triangular graph by weight. These basic solids compositions are listed in Table 2.

Table 2
SOLIDS COMPOSITION BY WEIGHT

| | | | |
|-------------------------|----|----|----|
| Teflon 7 | 16 | 16 | 16 |
| Bentone 38 | 1 | 1 | 1 |
| Acryloid B-72 (100%) | 71 | 59 | 47 |
| Pigment | 12 | 24 | 36 |

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It will be noted that the Teflon 7 and Bentone 38 contents by weight were held constant, with the variations being in the ratios of pigment to film-forming resin. This type of variation was found in earlier studies to provide maximum information about the system. Table 3 lists the solids content by weight and by volume for the 18 anti-seize compositions in this experimental program. The solvent used in milling and diluting these compositions was MIBK.

Steel panels were coated with each composition by both brushing and spraying for the salt-spray test. Coatings on steel panels were also applied by doctor blade for the sand abrasion tests and tape adhesion tests.

The sand abrasion test on the dry films was run according to Method 6191 (Fed. Test Method Std. No. 141). The tape test was run according to Method 6301 except that no water immersion was involved. A 24 hour dry-time and nominal 1 mil dry film thickness was used in each case. Table 4 gives the results of these tests along with pigment composition data for orientation purposes. The most noteworthy result is that all 18 samples showed good abrasion resistance with the sand abrasion test. All but four samples showed less than 5% film removal on the tape test. There are some slight trends in the adhesion and abrasion resistance. The resistance for sand abrasion decreases for 5 of the 6 pigments as pigment content increases from 12 to 36% (with a maximum at 24% in two cases, however); the adhesion improves for 4 of the 6 pigments as pigment content increases.

The brushed and sprayed panels were started in the standard salt-spray test (20% salt solution) in July. After 200 hours the 36 panels were removed and examined for: (1) general coating appearance, (2) rust in the score area, (3) film adhesion by the tape test, and (4) film adhesion by the knife test. Because a large proportion of the coated panels were in satisfactory condition, especially with regard to corrosion, the salt-spray test was allowed to continue to 400 hours and,

Table 3

FIL EXPERIMENTAL ANTI-SEIZE COMPOUNDS: SOLIDS COMPOSITIONS

| Pigment & Specific Gravity | | Basic zinc Chromate, 3.77 | | | | | | Chrome yellow medium 5.41 | | | | | | Zinc yellow I-469-D, 3.48 | | | | | |
|----------------------------|-------|---------------------------|--------|--------|--------|--------|--------|---------------------------|--------|--------|--------|-----|--------|---------------------------|--------|--------|--------|-----|--------|
| FIL Composition Number | | 27-2-1 | 27-2-2 | 27-2-3 | 27-2-4 | 27-2-5 | 27-2-6 | 27-2-7 | 27-2-8 | 27-2-9 | | | | 27-2-7 | 27-2-8 | 27-2-9 | | | |
| Ingredients Sp. Gr. | | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. |
| Teflon 7 | 2.16 | 16 | 9.98 | 16 | 11.10 | 16 | 12.50 | 16 | 10.11 | 16 | 11.43 | 16 | 13.14 | 16 | 9.95 | 16 | 11.01 | 16 | 12.34 |
| Bentone 38 | 1.80 | 1 | 0.75 | 1 | 0.83 | 1 | 0.94 | 1 | 0.76 | 1 | 0.85 | 1 | 0.99 | 1 | 0.75 | 1 | 0.83 | 1 | 0.93 |
| Acryloid B-72 (100%) | 1.126 | 71 | 84.98 | 59 | 78.53 | 47 | 70.45 | 71 | 86.10 | 59 | 80.87 | 47 | 74.06 | 71 | 84.67 | 59 | 77.91 | 47 | 69.51 |
| Pigment | -- | 12 | 4.29 | 24 | 9.54 | 36 | 16.11 | 12 | 3.03 | 24 | 6.85 | 36 | 11.81 | 12 | 4.63 | 24 | 10.25 | 36 | 17.22 |
| TOTAL | -- | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 |

| Pigment & Specific Gravity | | Basic lead silico chromate, 4.1 | | | | | | Basic lead chromate, 6.90 | | | | | | Strontium chromate, 3.89 | | | | | |
|----------------------------|-------|---------------------------------|---------|---------|---------|---------|---------|---------------------------|---------|---------|--------|-----|--------|--------------------------|---------|---------|--------|-----|--------|
| FIL Composition Number | | 27-2-10 | 27-2-11 | 27-2-12 | 27-2-13 | 27-2-14 | 27-2-15 | 27-2-16 | 27-2-17 | 27-2-18 | | | | 27-2-16 | 27-2-17 | 27-2-18 | | | |
| Ingredients Sp. Gr. | | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. | Wt. |
| Teflon 7 | 2.16 | 16 | 10.02 | 16 | 11.19 | 16 | 12.67 | 16 | 10.18 | 16 | 11.60 | 16 | 13.49 | 16 | 10.00 | 16 | 11.13 | 16 | 12.56 |
| Bentone 38 | 1.80 | 1 | 0.75 | 1 | 0.84 | 1 | 0.95 | 1 | 0.76 | 1 | 0.87 | 1 | 1.01 | 1 | 0.75 | 1 | 0.84 | 1 | 0.94 |
| Acryloid B-72 (100%) | 1.126 | 71 | 85.27 | 59 | 79.13 | 47 | 71.37 | 71 | 86.67 | 59 | 82.08 | 47 | 76.00 | 71 | 85.09 | 59 | 78.76 | 47 | 70.80 |
| Pigment | -- | 12 | 3.96 | 24 | 8.84 | 36 | 15.01 | 12 | 2.39 | 24 | 5.45 | 36 | 9.50 | 12 | 4.16 | 24 | 9.26 | 36 | 15.70 |
| TOTAL | -- | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 | 100 | 100.00 |

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Table 4. Sand Abrasion and Tape Adhesion Tests
on 18 Anti-Seize Compound Films

| <u>FIL Composition Number</u> | <u>Pigment Used</u> | <u>% Pigment by Wt. in Dry Film</u> | <u>Sand Abrasion Coefficient (liters/mil)</u> | <u>Tape Test, Approx. % Film Removed</u> |
|-----------------------------------|----------------------------|---|---|--|
| 27-2-1 | Basic zinc chromate | 12 | 12.4 | 20 |
| 27-2-2 | Basic zinc chromate | 24 | 12.7 | 5 |
| 27-2-3 | Basic zinc chromate | 36 | 10.3 | None |
| 27-2-4 | Chrome yellow medium | 12 | 13.4 | 3 |
| 27-2-5 | Chrome yellow medium | 24 | 14.2 | None |
| 27-2-6 | Chrome yellow medium | 36 | 16.1 | None |
| 27-2-7 | Zinc yellow | 12 | 13.9 | 90 |
| 27-2-8 | Zinc yellow | 24 | 13.7 | None |
| 27-2-9 | Zinc yellow | 36 | 7.9 | None |
| 27-2-10 | Basic lead silico-chromate | 12 | 10.9 | 10 |
| 27-2-11 | Basic lead silico-chromate | 24 | 14.8 | 75 |
| 27-2-12 | Basic lead silico-chromate | 36 | 11.9 | 5 |
| 27-2-13 | Basic lead chromate | 12 | 19.0 | 2 |
| 27-2-14 | Basic lead chromate | 24 | 12.2 | 1 |
| 27-2-15 | Basic lead chromate | 36 | 11.7 | 5 |
| 27-2-16 | Strontium chromate | 12 | 20.5 | None |
| 27-2-17 | Strontium chromate | 24 | 9.7 | 1 |
| 27-2-18 | Strontium chromate | 36 | 7.8 | 5 |

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Table 5

| 800 Hour Salt-Spray Corrosion Test Results | | | | | | Condition of Film | | | Condition of Substrate | | |
|--|----------------------------|------------------------------|---|--------------|------------------------------------|---|-------------------|-----------------------|--|-----------------------|--|
| FIL Composition Number | Pigment Used | % Pigment by Wt. in Dry Film | Method of Application and Film Thickness (Range Over Both Sides) (Mils) | No. of Coats | General Appearance of Coated Panel | Adhesion of Film (Water-Rinsed & Dried) Knife Test Rating | Rust In the Score | Blister Formation | Discoloration | General Rust | Pits and Localized Rust Spots |
| 27-2-1 | Basic zinc chromate | 12 | BRUSH 2.5-3.5 SPRAY 3-4 | 3 | F+ F | P P+ | M L-M | extensive slight | nil nil | nil nil | slight moderate |
| 27-2-2 | Basic zinc chromate | 24 | BRUSH 3-5 SPRAY 3.5-5 | 3 | G+ G | P+ F+ | H M-H | slight nil | nil nil | nil nil | nil slight |
| 27-2-3 | Basic zinc chromate | 36 | BRUSH 3.5-5.5 SPRAY 2.5-3.5 | 3 | G+ F- | E- E | H M-H | nil nil | nil moderate | nil slight | nil extensive |
| 27-2-4 | Chrome yellow medium | 12 | BRUSH 1.5-2.5 SPRAY 2.5-4 | 2 | F- F- | F- P+ | H M-H | extensive nil | nil nil | nil nil | nil ^b slight ^b |
| 27-2-5 | Chrome yellow medium | 24 | BRUSH 1.5-2.5 SPRAY 2-3 | 1 | F+ F- | F- P+ | M-H M-H | nil nil | slight ^a slight ^a | slight nil | nil ^b slight ^b |
| 27-2-6 | Chrome yellow medium | 36 | BRUSH 1.5-2.5 SPRAY 1.5-2 | 1 | G G- | F F- | M-H H | nil nil | nil slight ^a | nil nil | slight ^b slight ^b |
| 27-2-7 | Zinc yellow | 12 | BRUSH 2.5-3.5 SPRAY 2.5-4.5 | 2 | G F- | F+ F | H M-H | nil moderate | nil slight | nil slight | nil extensive |
| 27-2-8 | Zinc yellow | 24 | BRUSH 2.5-3.5 SPRAY 2-3.5 | 2 | G+ F+ | E E | L-M M-H | nil nil | nil nil | nil slight | nil extensive |
| 27-2-9 | Zinc yellow | 36 | BRUSH 2.5-3 SPRAY 1.5-3.5 | 2 | F P | G+ G | L-M M | nil nil | nil moderate | moderate extensive | extensive extensive |
| 27-2-10 | Basic lead silico-chromate | 12 | BRUSH 3-3.5 SPRAY 2.5-3.5 | 2 | E- P+ | P F- | M-H M-H | extensive moderate | nil slight | nil slight | nil ^c extensive |
| 27-2-11 | Basic lead silico-chromate | 24 | BRUSH 3-3.5 SPRAY 3.5 | 2 | E- F- | F F- | M M-H | moderate slight | nil slight | nil nil | nil ^c moderate |
| 27-2-12 | Basic lead silico-chromate | 36 | BRUSH 3-3.5 SPRAY 2-3 | 2 | G P+ | F F- | M-H M-H | slight slight | nil slight | slight nil | slight ^c extensive |
| 27-2-13 | Basic lead chromate | 12 | BRUSH 2-3 SPRAY 2-3 | 3 | F P+ | P+ F | M-H M-H | extensive moderate | nil slight | moderate moderate | slight extensive |
| 27-2-14 | Basic lead chromate | 24 | BRUSH 2-3 SPRAY 2-3 | 2 | F- P+ | P+ G | M-H M-H | extensive slight | nil nil | slight moderate | moderate extensive |
| 27-2-15 | Basic lead chromate | 36 | BRUSH 2.5-3.5 SPRAY 1.5 | 2 | F P | F- F- | M-H M-H | moderate slight | nil nil | slight moderate | nil extensive |
| 27-2-16 | Strontium chromate | 12 | BRUSH 2.5-3.5 SPRAY 1.5-3 | 2 | G+ F- | F- F | L M-H | slight moderate | nil nil | slight slight | nil ^c slight |
| 27-2-17 | Strontium chromate | 24 | BRUSH 2.5-3.5 SPRAY 2.5-3.5 | 2 | G+ F+ | F G | L-M H | slight nil | nil nil | nil nil | slight ^c moderate ^c |
| 27-2-18 | Strontium chromate | 36 | BRUSH 2.5-4 SPRAY 3.5-4 | 2 | F- F- | E- E- | M M-H | nil nil | nil nil | slight slight | extensive extensive |

E = Excellent
G = Good
F = Fair
P = Poor

H = Heavy
M = Moderate
L = Light

a - orange darkening around edges
b - scraped-off coating darker on under-side
c - thin waxy layer on surface (paraffin?)

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after further examinations, to 600 and 800 hours (more than 33 days) at which time it was concluded.

The panels were rinsed in tap water, blotted dry, and allowed to dry thoroughly in air before examining. The results of the examination of the 36 panels (18 brushed and 18 sprayed) are listed in Table 5. The panels were rated for general appearance, adhesion by knife test (the tape test was not suitable at this point because of corrosion products and, in some instances, waxy films on the coating surface), rust in the score mark, condition of the film (including blister formation and discoloration) and condition of substrate (including general rust and localized rust spots).

The 800 hour salt-spray test is a severe one and the panels as a whole fared remarkably well. In our analysis of the results, the effects of some of the coating factors were seen.

Table 6 is a comparison of corresponding brushed and sprayed panels.

Table 6

| | <u>Brushed Panels Better</u> | <u>Sprayed Panels Better</u> | <u>Tied</u> |
|----------------------------------|----------------------------------|----------------------------------|-------------|
| General Appearance | 16 | None | 2 |
| Pits and Localized Rust Spots | 15 | None | 3 |
| Knife Test Adhesion | 7 | 8 | 3 |
| Film Thickness | 8 greater | 7 greater | 3 |

It can be seen that brushing appears to be a substantially better application technique where general appearance and corrosion prevention are concerned. It is striking that in no case were the sprayed panels superior to the brushed panels in these properties. Difference in film

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thickness is seen not to be the causative factor in the superiority of the brushed panels. Application method appears to have no significant effect on adhesion properties.

Table 7 is a comparison of corresponding panels with the different pigment levels (12%, 24% and 36%).

Table 7

| | <u>12%</u> <u>Best</u> | <u>24%</u> <u>Best</u> | <u>36%</u> <u>Best</u> | <u>12-24%</u> <u>Tie</u> | <u>12-36%</u> <u>Ties</u> | <u>24-36%</u> <u>Tie</u> | <u>3-Way</u> <u>Tie</u> |
|----------------------------------|---------------------------|---------------------------|---------------------------|-----------------------------|------------------------------|-----------------------------|----------------------------|
| General Appearance | None | 5 | 2 | 3 | 1 | 1 | None |
| Pits and Localized Rust Spots | 3 | 2 | None | 3 | None | 1 | 3 |
| Knife Test Adhesion | None | 3 | 7 | None | None | 1 | 1 |

For general appearance the 24% level appears to be best. For prevention of localized rust spots the 12% and 12-24% levels appear to be best; no 36% samples ranked better than the comparable 12-24% samples. For adhesion a very distinct trend of improvement from low to high pigment content appears, with none of the 12% samples ranking as best. We consider this to be, to some degree, related to the blistering failures often noted with the low-pigment, high-resin compositions. (Note the strong correlation in Table 1.) Comparison of the adhesion test results for the compounds unexposed (Table 4) and after 800 hours in the salt-spray shows fairly good correlation for the effect of pigment content. Another factor in the 800 hour test is the relatively impervious nature of the low-pigment, high-resin film which can lead to osmotic pressure differentials and water blister formation. A conclusion of importance is that none of the pigments in this test overcome the water blister and adhesion loss problem associated with high resin content.

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Table 8 is a numerical rating of the systems by pigment type by summation using the following set of values: excellent or nil, 4; good or slight, 3; fair or moderate, 2; poor or extensive, 1. Plus or minus marks were accounted for by adding or subtracting 0.3 to the rating.

Table 8

| | Basic Zinc <u>Chromate</u> | Chrome Yellow <u>Medium</u> | Zinc <u>Yellow</u> | Basic Lead Silico <u>Chromate</u> | Basic Lead <u>Chromate</u> | Strontium <u>Chromate</u> |
|-------------------------------------|----------------------------------|-----------------------------------|-----------------------|--|----------------------------------|------------------------------|
| General Appearance | 15.6 (1) | 13.1 (5) | 13.3 (4) | 14.7 (2) | 9.3 (6) | 14.0 (3) |
| Pits and Localized Rust Spots | 17.0 (2) | 20.0 (1) | 12.0 (5) | 15.0 (3) | 10.0 (6) | 14.0 (4) |
| Knife Test Adhesion | 13.6 (3) | 9.7 (6) | 18.6 (1) | 10.1 (5) | 11.0 (4) | 16.1 (2) |
| Total | 46.2 (1) | 42.8 (4) | 43.9 (3) | 39.8 (5) | 30.3 (6) | 44.1 (2) |

The basic zinc chromate panels ranked first for general appearance (rankings in parentheses). The chrome yellow medium panels ranked first for freedom from pits and rust spots, but were poorest in over-all adhesion. The zinc yellow panels ranked first in knife test adhesion but were relatively poor in protection from pitting and rust spots. An important result is that the best pigment in over-all ranking was basic zinc chromate, which has been the prime pigment under consideration in our program for a period of years. This, in a sense, confirms our earlier choice. The second ranking pigment in this 800 hour program was strontium chromate; this is of some interest as it had a higher adhesion ranking than the basic zinc chromate.

2.15 Aberdeen Test Results

The superiority of the low Acryloid-high chromate formulations over the high Acryloid types was supported in part toward the end of this project by the results of the Aberdeen field tests with the high-Acryloid compound FIL 15-13-2 on an M-59 vehicle, in which the compound was found to be unsatisfactory. (This is in contrast with the "satisfactory" results obtained earlier in a test with the lower-Acryloid FIL 92-46-1 at Yuma.) The disassembled parts were examined and the test results were discussed with R. Ammon (the test report author). The "unsatisfactory" conclusion was concurred with.

One outcome of these field tests, when examined in light of laboratory test results, is (1) that the drying time earlier specified is insufficient, and (2) that the higher-adhesion compound (FIL 92-46-1) should be used. This conclusion is based on the results at many points of application in which there was reported "no evidence of compound remaining" or "compound had flaked loose." It is our feeling that the modified 92-46-1 compound applied using the extended drying times would serve satisfactorily under equivalent 3000 mile field testing conditions.

The considerations set forth in the last part of Section 2.9 would be pertinent in reference to discussion of high values of required disassembly forces in large areas of anti-seize films involved in pure shear during a removal process.

2.16 Soil Burial Test Results

The soil burial test of standard-torqued nut-bolt combinations and coated steel plates was concluded after a total burial period of 434 days (a little over 14 months). The soil used was a high organic-matter top soil, which was initially adjusted to a pH of 9 with sodium carbonate; also water and sodium chloride were added. Examination of the specimens was made after four months burial, with the samples then returned to the mud. The pH of the mud after 434 days was 8.5, the same as that at four months.

The coatings in the test were as follows:

- (1) Control (no coating)
- (2) Molykote M-88 (molybdenum disulfide in a resin binder)
- (3) Acryloid B-72 (from a solution in toluene)
- (4) FIL 92-46-1 (Teflon-Acryloid-basic zinc chromate composition sent to Yuma)
- (5) FIL 15-10-15 (high-Acryloid composition, slight modification of which was sent to Aberdeen as 15-13-2)
- (6) FIL 15-10-16 and
- (7) FIL 15-10-17 (compounds having higher Teflon content than FIL 15-10-15).

Figure 3 illustrates the panels (left to right, top row first) in the order listed above. The control panel (no coating) was eaten away completely for about 18% of its area, the attack being worst at the lower edge of the panel as supported vertically in the soil. Severe etching took place elsewhere on the panel.

A similar attack took place on the panel coated with a half mil of Molykote M-88, though less severe (8% area completely eaten away). The panel with 1 mil Acryloid B-72 coating was in better condition. There was heavy blistering along the lower edge, with red rust formation. Most of the area was in fair condition.

The panels coated with the four anti-seize compounds (2-4 mils) were about equivalent to each other in appearance and somewhat superior to the Acryloid-coated panel. A number of rust-filled blisters were present on each panel; however, the attack seemed to be confined to a surface attack, without pits. Most of the surface under the anti-seize compound films was completely protected. In general, the four anti-seize compounds are rated as satisfactory in this severe test.

The nut-bolt test has provided, most importantly, a measure of the anti-seize properties of the compounds under severe corrosive conditions.

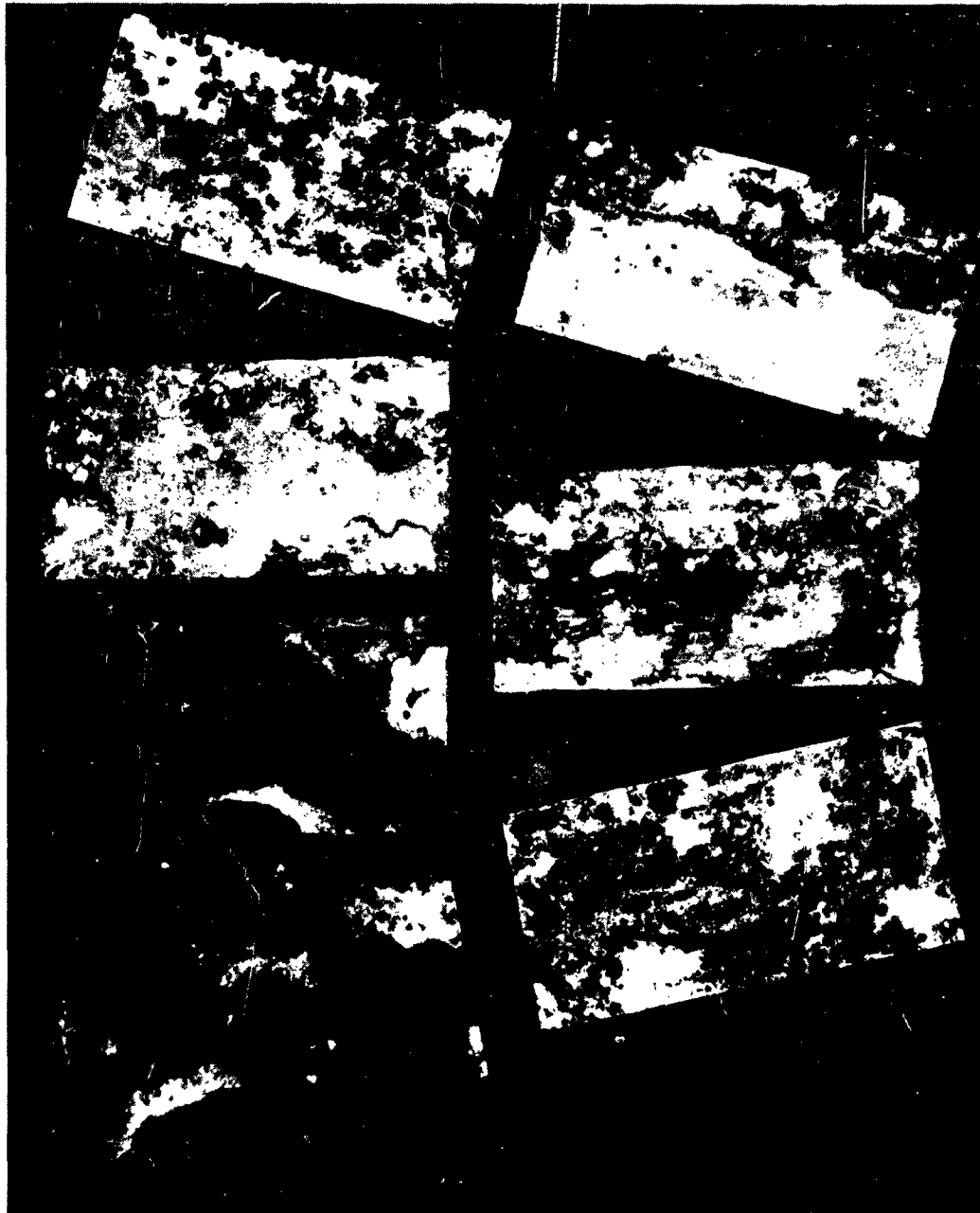


FIG. 3 SOIL BURIAL TEST SPECIMENS AFTER 14 MONTHS EXPOSURE

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After removal from the wet test soil, the bolts were rinsed in tap water and allowed to dry. There was no great rust on any bolt, and very little visible corrosion on the exposed surfaces. (The alkaline soil tends to cleanly etch exposed surfaces without rust formation.)

The torque to loosen each nut-bolt combination was measured with a torque wrench. There were three samples buried for each anti-seize compound so as to permit a study of reproducibility. All bolts were tightened 14 months earlier to a uniform 120 ft-lbs. Table 9 lists the loosening torques for each sample.

We note that each of the anti-seize compounds produces substantially lower resistance to loosening than does no treatment. (This does not hold true for newly-assembled nuts and bolts, as we had seen in much earlier studies.) Another point of note is that all of the Teflon and chromate-modified compounds were superior to the unmodified Acryloid B-72; this indicates that the Teflon-chromate addition indeed enhances the anti-seize properties.

The compound producing the lowest average maximum loosening torques is FIL 92-46-1, the material sent to Yuma for field tests. This superiority (though slight) is another indication that this formulation is more effective than the higher-Acryloid types, such as FIL 15-10-15, 16 and 17.

3. CONCLUSION

The experimental programs completed on this contract have served to optimize the performance of the protective coating type anti-seize composition. The basic 3-component formulation consisting of a soft acrylic resin (Acryloid B-72), tetrafluoroethylene powder (Teflon 7) and a corrosion inhibiting pigment was arrived at as described in the final report for the previous project (Contract No. DA-36-034-ORD-2434).

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Table 9

LOOSENING TORQUES FOR
NUT-BOLT COMBINATIONS AFTER 14 MONTH SOIL BURIAL TEST

| <u>Test Sample No.</u> | <u>Anti-seize Compound Used</u> | <u>Bolt Designation</u> | <u>Maximum Torque to Loosen (ft-lb)</u> | <u>Average Maximum Loosening Torque (ft-lb)</u> |
|--------------------------------|--|-----------------------------|---|---|
| 1 | None (control) | 1A | 145 | 138 |
| | | 1B | 130 | |
| | | 1C | 140 | |
| 2 | Molykote M-88 (MoS2 in resin binder) | 2A | 95 | 95 |
| | | 2B | 92 | |
| | | 2C | 98 | |
| 3 | Acryloid B-72 (from solution) | 3A | 115 | 105 |
| | | 3B | 103 | |
| | | 3C | 96 | |
| 4 | FIL 92-46-1 | 4A | 92 | 93 |
| | | 4B | 93 | |
| | | 4C | 93 | |
| 5 | FIL 15-10-15 | 5A | 94 | 96 |
| | | 5B | 100 | |
| | | 5C | 95 | |
| 6 | FIL 15-10-16 | 6A | 101 | 95 |
| | | 6B | 87 | |
| | | 6C | 98 | |
| 7 | FIL 15-10-17 | 7A | 93 | 94 |
| | | 7B | 96 | |
| | | 7C | 93 | |

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The choice of basic zinc chromate as the optimum pigment was confirmed by testing program. The ratio of pigment to resin of the FIL 92-46-1 composition was seen to lie in the best region for over-all properties, and to be significantly better than the ratios in high-Acryloid compositions for adhesion properties. The drying time required for optimum performance was seen to be considerably greater than suspected at the beginning of the program. Other important findings relate to best procedures for dispersion and to the use of a high-boiling strong solvent to reduce brushmarks in application.

We feel that the FIL 92-46-1 anti-seize compound, the composition of which is shown in Appendix A, when applied according to the instructions in Appendix B, will be generally satisfactory in anti-seize application for tank vehicles.



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Project Engineer

Approved by:



Edmund Thelen, Manager •
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Director of Laboratories

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APPENDIX A

FIL 92-46-1 BASE STOCK COMPOSITION

| | <u>% by Weight</u> | <u>% by Weight in Solids</u> | <u>Parts on 100 Parts Resin, by Weight</u> | <u>% by Volume</u> |
|----------------------|------------------------|--------------------------------------|--|------------------------|
| Acryloid B-72 (100%) | 28.55 | 52.0 | 100.00 | 29.80 |
| Basic zinc chromate | 15.57 | 28.4 | 54.54 | 4.87 |
| Teflon 7 powder | 8.78 | 16.0 | 30.75 | 4.70 |
| Bentone 34 | 2.00 | 3.6 | 7.01 | 0.63 |
| TOTAL SOLIDS | 54.90 | 100.0 | 192.30 | 40.00 |
| MIBK | 45.10 | | 157.97 | 60.00 |
| TOTAL | 100.00 | | 350.27 | 100.00 |

Mixing Procedure: The Teflon powder is stirred into the MIBK. The Acryloid B-72 is then dissolved in this slurry. The basic zinc chromate and the Bentone 34 are mixed together, then stirred into the liquid portion. The total mix is given three passes on a tight three-roll mill, then pressure filtered through a 100 mesh screen. Adjustment to 54.9% solids is made with MIBK.

The diluting solvent is a mixture of 50% ethylene glycol monoethyl ether, 20% ethyl alcohol and 30% MIBK by weight. For ordinary brushing application it is mixed 1:1 by volume with the above base stock.

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APPENDIX B

SURFACE PREPARATION PRIOR TO APPLICATION OF EXPERIMENTAL
DETROIT ARSENAL--FRANKLIN INSTITUTE ANTI-SEIZE COMPOUND

The surfaces to be coated must be free of grease, oil, loose rust, dirt, etc.

To insure suitable surface conditions the following procedure is to be used:

- (1) Any bulk dirt, grease, etc., is wiped off with a rag.
- (2) The metal surface is wire brushed thoroughly to remove all loose or flaky rust.
- (3) The metal surface is then washed and rubbed vigorously with a clean cloth soaked with stabilized trichloroethylene (Military Specification MIL-T-7003). This is repeated until no further visible grease, loose rust, etc., is removed thereby. The surface is then dried with a clean oil-free cloth such as cheese cloth, or is air dried in clean air. The dry surface is then ready for application of the anti-seize compound.

APPLICATION INSTRUCTIONS FOR THE ANTI-SEIZE COMPOUND

The base stock is designated as FIL 92-46-1. The diluent is a 50:30:20 mixture by weight of ethylene glycol monoethyl ether (Cellosolve), MIBK and ethyl alcohol. The application procedure is as follows:

- (1) Stir the base stock in its container until it appears completely homogeneous (no bottom settling and no thin or clear liquid on top).
- (2) Mix one part by volume of the base stock to one part by volume of the diluent and stir until completely homogeneous. This will produce the finished anti-seize compound of consistency suitable for brushing application from about 70°F to 100°F.

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(3) The diluted anti-seize compound is to be applied with a high-quality paint brush of about 1"--1-1/2" width. The material should be brushed as a single coat thinly and uniformly on each of the two surfaces to be protected for any given mating fit. Extend the coatings beyond the mating area in all cases. The coating on the two halves should be allowed to dry for 48 hours before assembly of the parts.

SAFETY PRECAUTIONS FOR USE WITH THE EXPERIMENTAL
DETROIT ARSENAL--FRANKLIN INSTITUTE ANTI-SEIZE COMPOUND

Care should be taken to keep the anti-seize base stock and the diluent away from fire, heat, or open lights as the solvent used are moderately flammable. The tag open cup flash point of MIBK is 98°F.

The trichloroethylene recommended for cleaning the surfaces prior to coating with anti-seize compound is among the safest of the chlorinated solvents. The only important precaution is to work in a ventilated area.

APPENDIX C

SPECIFICATION ANTI-SEIZE COMPOUND,
POLYTETRAFLUOROETHYLENE AND ZINC CHROMATE FILLED RESIN TYPE

1. SCOPE

- 1.1 Scope - This specification covers the requirements for a lacquer-type corrosion-inhibiting anti-seize compound, containing powdered polytetrafluoroethylene and zinc chromate, primarily intended for use on contiguous parts in tank vehicles.
- 1.2 Classification - This specification covers one grade of anti-seize compound in yellow, characteristic of the basic zinc chromate pigment, and other such colors as may be required.

2. APPLICABLE DOCUMENTS

- 2.1 The following publications, of the issue in effect on the date of invitation for bids, shall form a part of this specification to the extent specified herein:

2.1.1 Specifications

Federal

Test Method Std. No. 141-Paint, Varnish, Lacquer,
and Related Materials; Methods of Inspection, Sampling
and Testing

TT-M-268-Methyl Isobutyl Ketone (MIBK)

Military

MIL-T-7003-Trichloroethylene

MIL-P-15328-Basic Zinc Chromate

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3. REQUIREMENTS

3.1 Materials - The ingredient materials used in the manufacture of this product shall be of high quality, suitable for the purpose, and shall conform to applicable requirements as specified herein. Ingredient materials conforming to contractor's specification may be used provided the specifications are acceptable to the Government and contain provisions for adequate tests. The use of the contractor's specifications will not constitute waiver of Government inspection.

3.2 Composition - The composition of the anti-seize compound shall be as specified in Table I.

Table I. COMPOSITION, PERCENT BY WEIGHT

| <u>Ingredient</u> | <u>Percent Minimum</u> | <u>Percent Maximum</u> |
|---|----------------------------|----------------------------|
| Nonvolatile Portion | 53.0 | 56.5 |
| Acryloid B-72 (100%)* | 27.0 | 30.0 |
| Basic zinc chromate (MIL-P-15328) | 15.0 | 16.5 |
| Teflon 7** (tetrafluoroethylene polymer, 35 micron powder) | 8.5 | 9.5 |
| Volatile Portion | | |
| Methyl isobutyl ketone | 43.5 | 47.0 |

* Rohm and Haas Company

** DuPont

Diluent: 50:30:20 mixture of ethylene glycol monomethyl ether,
MIBK and ethanol.

3.3 Ingredients - All ingredients shall conform to applicable Government specifications.

3.4 Physical Properties

- 3.4.1 Condition in Container - The anti-seize compound shall be free from skins and lumps, and shall be capable of being easily mixed to a smooth homogeneous condition, both in the original container and when reduced as specified in 3.5.1.
- 3.4.2 Appearance - The appearance of the anti-seize compound shall be uniform and homogeneous when examined according to Method 4262 (Fed. Test Method Std. No. 141). There shall be no trace of grit or of rough particles substantially in excess of the Teflon 7 particle size (35 microns).
- 3.4.3 Coarse Particles - Coarse particles shall not exceed 0.1 percent by weight retained when run according to Method 4092, Fed. Test Method Std. No. 141, using a sieve No. 100 and MIBK as the washing solvent.
- 3.4.4 Weight per Gallon - The weight per gallon shall be from 8.4-8.6 pounds, and shall be determined by Method 4184, Fed. Test Method Std. No. 141.
- 3.4.5 Viscosity - The viscosity of the anti-seize compound when reduced by the addition of 1 part of diluent (Sec. 3.2) to 1 part of package material by volume shall be not less than 20 seconds and not more than 30 seconds when measured in a No. 2 Zahn cup at 22°C.*

*These values depend to an extent on the exact milling technique and would probably need revision for large scale mill batches.

3.4.6 Storage Stability - After 1 year of storage under the conditions specified in Method 4142, Fed. Test Method Std. No. 141, a full container of anti-seize compound package material shall show no skinning, livering, curdling, hard settling, or caking. After the above storage, the anti-seize compound shall conform to all tests of this specification; some increase in viscosity will be permitted provided that the anti-seize compound exhibits satisfactory working properties.

3.5 Film Properties

3.5.1 Application - Packaged anti-seize compound, reduced 1 volume of anti-seize compound to 1 volume of diluent (Sec. 3.2) conforming to Specification TT-M-268, shall be a freely working product, and shall be of a consistency suitable for brushing application. The 1:1 dilution shall be used in the film property tests.

3.5.2 Metal Adhesion - Anti-seize compound films shall be applied by doctor blade to a 1 mil dry film thickness over trichloroethylene - cleaned steel panels and shall be air-dried for 24 hours. The adhesion of the anti-seize compound film shall be determined in accordance with Method 6304, Fed. Test Method Std. No. 141, entitled "Adhesion (Knife Test)." The film shall be tough and well bonded.

3.5.3 Metal Adhesion - Anti-seize compound films shall be applied by doctor blade to a 1 mil dry film thickness over trichloroethylene - cleaned steel panels and shall be air-dried for 24 hours. The adhesion of the anti-seize compound shall be determined by the rapid-removal

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masking tape test; to be run by Method 6301, Fed. Test Method Std. No. 141, except that no water immersion is to be used. The coating adhesion shall be deemed satisfactory if the coating is not removed for more than 1/8" from the score-mark.

- 3.5.4 Abrasion Resistance - Anti-seize compound films shall be applied by doctor blade to a 2-3 mil wet film thickness over trichloroethylene - cleaned steel panels and shall be air-dried for 24 hours. The abrasion resistance (falling sand) shall be determined by Method 6191, Fed. Test Method Std. No. 141. The coating shall have an abrasion coefficient of greater than 3.5 liters per mil of dry film thickness.
- 3.5.5 Drying Time - Films of anti-seize compound diluted 1:1 with MIBK to plate glass panels by doctor blade to a 0.0015 inch wet film thickness. Drying time shall be determined by Method 4061, Fed. Test Method Std. No. 141, Section 3.2, entitled "Set-to-Touch," and Section 3.6, entitled "Dry Through." The film shall be set-to-touch in not more than 6 minutes. The film shall be dry through in not more than 15 minutes.

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